

CLAIMS

1. A method for synthesis of an oxime by ammoximation of a carbonyl compound, comprising:

5 (i) reacting a carbonyl compound containing 6 to 20 C atoms with ammonia and hydrogen peroxide in the presence of

a) an organic solvent that is a1) at least partly water-soluble, a2) stable under ammoximation conditions, a3) has a boiling point of higher than 100°C and/or is capable of forming a two-phase azeotrope with water, and

10 b) a titanium-containing heterogeneous catalyst,

to obtain a reaction mixture containing said oxime,

(ii) separating the catalyst from the reaction mixture,

(iii) crystallizing the oxime and separating the crystallized oxime from the reaction mixture, thereby obtaining crystallized oxime and a mother liquor,

15 (iv) if the mother liquor is a two-phase system in which one of the phases is an aqueous phase, then water is removed from the mother liquor in the form of an aqueous phase, and

(v) distilling off water or a water-containing two-phase azeotrope from the mother liquor, while the distillation bottoms and, optionally, a predominantly organic phase of the azeotrope are recycled to stage (i).

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2. The method according to claim 1, wherein said solvent is an alcohol containing 2 to 6 C atoms.

3. The method according to claim 1, wherein said solvent is n-butanol.

4. The method according to claim 1, wherein the crystallizing of the oxime proceeds at a temperature ranging from -40 to +60°C.

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5. The method according to claim 1, wherein a linear or cyclic ketone containing 6 to 12 C atoms is ammoximated in the presence of n-butanol as solvent.

6. The method according to claim 1, wherein the ammoximation is performed in the presence of a catalyst containing titanium silicalite.

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7. The method according to claim 1, wherein the ammoximation is performed in the presence of a solid acid cocatalyst, a soluble ammonium salt or a combination of a solid acid cocatalyst and a soluble ammonium salt.

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8. The method according to claim 1, wherein the ammoximation is performed continuously in one or more fixed-bed reactors connected in series and operated in trickling-bed or bubbling mode at 25 to 150°C.

9. The method according to claim 2, wherein said alcohol containing 2 to 6 C atoms is a monohydric alcohol containing 4 to 6 C atoms or a dihydric alcohol.

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10. The method according to claim 1, wherein the crystallizing of the oxime proceeds at a temperature ranging from -10 to +30°C.

11. The method according to claim 1, wherein a linear or cyclic ketone containing 8 to 12 C atoms is ammoximated in the presence of n-butanol as solvent.

12. The method according to claim 1, wherein the ammoximation is performed in the presence of a solid acid cocatalyst selected from the group consisting of Al_2O_3 , TiO_2 , ZrO_2 and acid zeolites.

13. The method according to claims 1, wherein the ammoximation is performed in the presence of a soluble ammonium salt selected from the group consisting of ammonium nitrate, hydroxylammonium nitrate, ammonium phosphates, ammonium pyrophosphates, ammonium salts of carboxylic acids and ammonium stannates.

14. The method according to claim 1, wherein the ammoximation is performed additionally in the presence of a combination of a) a solid acid cocatalyst selected from the group consisting of Al_2O_3 , TiO_2 , ZrO_2 and acid zeolites, and b) a soluble ammonium salt selected from the group consisting of ammonium nitrate, hydroxylammonium nitrate, ammonium phosphates, ammonium pyrophosphates, ammonium salts of carboxylic acids and ammonium stannates.

15. The method according to claim 1, wherein the ammoximation is performed continuously in one or more fixed-bed reactors connected in series and operated in trickling-bed or bubbling mode at 50 to 125°C.

5 16. The method according to claim 1, wherein the distillation bottoms have a water content of from 0.01 to 5 wt%.

17. The method according to claim 1, wherein said titanium-containing heterogeneous catalyst is in the form of pellets.

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18. The method according to claim 1, wherein a molar ratio of ammonia to hydrogen peroxide is from 1.5 to 3.

19. The method according to claim 1, wherein a molar ratio of hydrogen peroxide to
15 carbonyl compound is from 0.5 to 1.5.

20. The method according to claim 1, wherein said ammoximation proceeds at a pressure of from 1 to 15 bar.